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TITLE: Magnesium oxide material for plasma displays has specific purity and contains predetermined amounts of calcium oxide and silicon oxide

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ABSTRACTED-PUB-NO: JP2000103614A

BASIC-ABSTRACT:

NOVELTY - The MgO material has a purity of 99.6 weight% (wt.%) and contains 3000 ppm or less of calcium oxide (CaO) and 2000 ppm or less of silicon oxide (SiO<sub>2</sub>) in a weight ratio of 4 or less. The MgO crystal grain have an average diameter of 500 mu m or less and density of 3.45 g/cm<sup>3</sup> or more.

DETAILED DESCRIPTION - INDEPENDENT CLAIMS are also included for the following: (i) manufacture of MgO material which involves electromelting MgO material having of purity 95 wt.% of more, (ii) plasma displays wherein a protective layer is formed on at least one side of a dielectric or fluorescent layer.

USE - For use as a protective layer for a dielectric and/or fluorescent material of a plasma display (claimed).

ADVANTAGE - Impurity content of CaO is controlled, transparency of MgO to ultraviolet region significantly improved. The MgO material has reduced hydration and improves productivity of plasma display.

CHOSEN-DRAWING: Dwg.0/0

TITLE-TERMS: MAGNESIUM OXIDE MATERIAL PLASMA DISPLAY SPECIFIC PURE CONTAIN  
PREDETERMINED AMOUNT CALCIUM OXIDE SILICON OXIDE

DERWENT-CLASS: E33 L03 V05

CPI-CODES: E34-B01; L03-C04;

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Chemical Indexing M3 \*01\*

Fragmentation Code

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C807 M411 M720 M903 M904 M910 N120 N163 Q454

Specific Compounds

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## 【特許請求の範囲】

【請求項1】プラズマディスプレイの誘電体及び／又は蛍光体のMgO保護層に用いるMgO材料であって、

(1) MgO純度99.6重量%以上、(2) CaO含有量3000ppm以下、SiO<sub>2</sub>含有量2000ppm以下であって、かつ、CaO/SiO<sub>2</sub>重量比4以下、(3) 平均結晶粒径500μm以上及び(4) 密度3.45g/cm<sup>3</sup>以上であることを特徴とするプラズマディスプレイ用MgO材料。

【請求項2】Fe、Cr、V、Cu、Ni、Zr及びLaの含有量合計が酸化物として1000ppm以下である請求項1記載のプラズマディスプレイ用MgO材料。

【請求項3】請求項1又は2に記載のプラズマディスプレイ用MgO材料の製造方法であって、MgO純度95重量%以上であるMgO原料を電融することを特徴とする製造方法。

【請求項4】プラズマディスプレイの誘電体及び蛍光体の少なくとも一方における保護層が請求項1又は2に記載のプラズマディスプレイ用MgO材料を原料として形成されたプラズマディスプレイ。

## 【発明の詳細な説明】

## 【0001】

【発明の属する技術分野】本発明は、プラズマディスプレイ（モノクロプラズマディスプレイ及びカラープラズマディスプレイの双方を含む）の誘電体及び／又は蛍光体の保護層に用いられるプラズマディスプレイ用MgO材料及びその製造方法ならびにプラズマディスプレイに関する。

## 【0002】

【従来技術】近年、輝度、発光効率等の性能に優れ、しかもテレビ表示のための表示品質も十分なプラズマディスプレイ（PDP）が市販され、待望の壁掛け型テレビの実現化にも期待が高まりつつある。

【0003】従来から使用されている電子銃励起によるブラウン管タイプのディスプレイ（CRT）ではブラウン管容積、表示画面の制約、輝度等に種々の問題を残しており、これらの問題を解消する新たな方式又は構造のディスプレイが検討され、近年になってようやくPDPが注目されるに至った。

【0004】この従来のCRTでは電子銃から放出された電子線が直接蛍光体を励起発光させているのに対し、PDPでは個々のセル内に存在する蛍光体を放電（紫外線）励起して発光させる。

【0005】PDPは、そのカラー表示方式として対向電極構造（対向型）と面放電構造（面放電型）に大別される。対向電極構造は、少なくとも一方のガラス基板上の電極近傍に蛍光体を塗布し、放電による紫外線照射により励起発光される。この場合、交流電圧の印加により蛍光体がスパッタリング（イオン衝撃）を受け、これが蛍光体の寿命に大きな影響を与えることが知られてい

る。そこで、蛍光体をスパッタリングから守るため、このスパッタリングに強いカソード材としてMgO保護層が蛍光体上に形成されている。一方、面放電構造では、イオン衝撃を受けない裏面ガラス基板上に予めMgO保護層を被覆した蛍光体を塗布し、放電に伴う紫外線照射によって励起発光させる。

【0006】このように、PDP（特にAC型）においては、MgO保護層はきわめて重要な役割を果たしている。ところが、現状ではこのMgO保護層を形成するためのMgO材料（原料）の特性に関する研究はほとんどなされていない。

## 【0007】

【発明が解決しようとする課題】PDPの保護層の形成方法としては、一般には電子ビーム蒸着法が採用されている。具体的には、高真空状態に維持されたチャンバーにおいて、MgO原料に電子ビームが照射されて揮発したMgOをガラス基板上に予め形成された誘電体上又は蛍光体上に析出させ、成膜することにより保護層が形成される。

20 【0008】しかしながら、従来のMgO原料では、MgO自体（特に表面部分）が水和しやすいという問題がある。すなわち、表面部分が水和したMgO原料を電子ビーム蒸着の原料ソースとして用いれば、目的真空度に到達するまでに長時間を要し、生産性に悪影響を及ぼしかねない。これに対し、予めMgO原料から水分を除去することも考えられるが、いったん水和してMg(OH)<sub>2</sub>が生成されると500℃以上という高温で加熱しなければ水分を完全に除去することができない。このため、特にPDP保護層に用いるMgO原料においては、保管状態を厳密に管理しなければならず、また保管期間の制約もある。

【0009】他方では、このような原料ソースを用いて形成されたMgO保護層の品質自体にも何らかの支障をもたらすことがある。

【0010】このように、MgO保護層の形成において、MgOの水和はPDPの生産性の低下、製造コストの上昇、あるいはPDPの品質低下等を招く原因となっており、これを解消することがPDPの製造において急務とされている。

40 【0011】従って、本発明は、かかる従来技術の実情に鑑みてなされたものであり、特に、水和が抑制ないしは防止されたMgO材料を提供することを主な目的とする。

## 【0012】

【課題を解決するための手段】本発明者は、従来技術の問題点に鑑みて鋭意研究を重ねた結果、特定の組成及び構造を有するMgO材料が上記目的を達成できることを見出し、ついに本発明を完成するに至った。

50 【0013】すなわち、本発明は、下記のプラズマディスプレイ用MgO材料及びその製造方法ならびにプラズ

マディスプレイに係るものである。

【0014】1. プラズマディスプレイの誘電体及び／又は蛍光体のMgO保護層に用いるMgO材料であって、(1) MgO純度99.6重量%以上、(2) CaO含有量3000ppm以下、SiO<sub>2</sub>含有量2000ppm以下であって、かつ、CaO/SiO<sub>2</sub>重量比4以下、(3) 平均結晶粒径500μm以上及び(4) 密度3.45g/cm<sup>3</sup>以上であることを特徴とするプラズマディスプレイ用MgO材料。

【0015】2. 上記第1項に記載のプラズマディスプレイ用MgO材料の製造方法であって、MgO純度95重量%以上であるMgO原料を電融することを特徴とする製造方法。

【0016】3. プラズマディスプレイの誘電体及び蛍光体の少なくとも一方における保護層が上記第1項に記載のプラズマディスプレイ用MgO材料を原料として形成されたプラズマディスプレイ。

【0017】なお、本発明において「ppm」は、重量割合(重量ppm)を示す。

【0018】

【発明の実施の形態】まず、本発明のMgO材料は、プラズマディスプレイの誘電体及び／又は蛍光体のMgO保護層に用いる原料(原料ソース)である。

【0019】本発明のMgO材料のMgO純度は通常99.6重量%以上、好ましくは99.7重量%以上とすれば良い。MgO純度が99.6重量%未満である場合は耐水和性が低下したり、あるいは保護膜の光学特性が低下するおそれがある。

【0020】MgO材料中のCaO含有量は通常3000ppm以下(好ましくは2000ppm以下)、SiO<sub>2</sub>含有量は通常2000ppm以下(好ましくは1500ppm以下)である。CaO含有量が3000ppmを超える場合には、MgO材料の耐水和性が低下する。SiO<sub>2</sub>含有量が2000ppmを超える場合には、PDP保護層を形成する際にSi<sup>4+</sup>イオンがMg<sup>2+</sup>イオンと置換したときに陽イオン欠損を生じ、MgO保護層の光吸収端が長波長側にシフト(励起光である紫外線を蛍光体のMgO保護層が吸収)し、蛍光体の発光輝度を低下させるおそれがある。

【0021】さらに、本発明では、CaO/SiO<sub>2</sub>重量比が通常4以下、好ましくは3.5以下とする。CaO/SiO<sub>2</sub>重量比が4を超える場合には、MgO材料の耐水和性が低下する。

【0022】また、MgO材料における平均結晶粒径は通常500μm以上、好ましくは1000μm以上とする。MgO材料における密度は通常3.45g/cm<sup>3</sup>以上、好ましくは3.48g/cm<sup>3</sup>以上とする。平均結晶粒径が500μm未満の場合及び密度が3.45g/cm<sup>3</sup>未満の場合には、大気中の水分との接触面積が全体として大きくなり、耐水和性を低下させることがあ

る。平均結晶粒径は、例えばSiO<sub>2</sub>含有量を制御したり、あるいは電融後における冷却速度の調整(例えば、熱容量の大きな大型電融炉にて溶融)等により適宜調節できる。また、上記密度は、原料を溶融する程度、電融品の選別等により適宜調節することができる。

【0023】なお、平均結晶粒径の上限は特に限定されず、従ってMgO単結晶も用いることができる。一方、密度の上限についても特に制限されず、理論密度(3.58g/cm<sup>3</sup>)まで高めることができる。

【0024】本発明のMgO材料では、Fe、Cr、V、Cu、Ni、Zr及びLaの含有量合計が酸化物として(すなわち、Fe<sub>2</sub>O<sub>3</sub>、CrO<sub>2</sub>、V<sub>2</sub>O<sub>5</sub>、CuO、NiO、ZrO<sub>2</sub>及びLa<sub>2</sub>O<sub>3</sub>として)通常1000ppm以下、特に700ppm以下とすることが好ましい。これらの元素(以下「遷移金属元素」という)の含有量合計を上記範囲に制御することによって、特に、このMgO材料を用いて形成されたMgO保護層が紫外～可視域にわたって安定した透明性をより効果的に発現することができる。遷移金属元素の総量を上記範囲に制御するためには、例えばMgO原料(組成)を選択したり、あるいはMgO原料の粉碎時等に脱鉄処理を施すことによって実施することができる。脱鉄処理は、例えば酸洗浄、磁力選鉱機による処理等によって実施することができる。

【0025】本発明MgO材料の製造方法は、基本的には上記範囲内のものが得られる限りは特に限定されないが、特にMgO純度95重量%以上であるMgO原料を電融することにより製造することが好ましい。かかる電融法によれば、上記所定の平均結晶粒径及び密度の範囲内に制御しやすく、しかもMgO純度95重量%程度のMgO原料を用いることができるという点で焼結法に比べて有利である。

【0026】MgO原料のMgO純度は通常95重量%以上、特に98重量%以上であることが好ましい。電融法を採用する本発明方法では、電融(溶融)によって、不純物が蒸発したり、あるいは不純物が電融MgOの周辺部に移動して偏在することから実質的に電融MgOのみを比較的容易に選別できるので、原料MgOよりも純度の高いMgOを得ることが可能となる。そして、選別された電融MgOをさらに電融及び選別という一連の工程を繰り返すことにより、より高純度化を図ることができ、このような態様も本発明の製造方法に包含される。従って、原料MgOとしてMgO純度が比較的低いものを用いる場合には、所定のMgO純度ならびに所定のCaO含有量、SiO<sub>2</sub>含有量及びCaO/SiO<sub>2</sub>重量比となるまで上記工程を繰り返し実施することが好ましい。

【0027】本発明の製造方法においてCaO含有量、SiO<sub>2</sub>含有量及びCaO/SiO<sub>2</sub>重量比は、例えば上記工程を繰り返すことにより制御できるほか、CaO、

SiO<sub>2</sub>等を積極的に添加することによって制御することができる。これらCaO、SiO<sub>2</sub>等は市販品をそのまま用いることができる。

【0028】本発明の製造方法における電融の操作条件、電融装置等は公知の電融方法で採用されている条件、装置等をそのまま採用することができる。例えば、所定のMgO原料をアーク炉に充填し、炭素電極にてアーク電流を通電することにより溶融し、溶融が完了した後に冷却（徐冷）を行い、得られた塊状物から電融MgOを選別して取り出せば良い。

【0029】本発明のPDPは、プラズマディスプレイの誘電体及び蛍光体の少なくとも一方におけるMgO保護層が本発明プラズマディスプレイ用MgO材料を原料（原料ソース）として形成されたものである。MgO保護層の形成方法は、公知の薄膜形成技術をそのまま適用することができ、例えば誘電体又は蛍光体層を形成した後、電子ビーム蒸着法等の方法を用いてMgO薄膜を作製することができる。

【0030】MgO保護層の厚さは、PDPの形式・方式、保護層の形成対象（誘電体、蛍光体等）最終製品の用途等に応じて適宜設定すれば良い。

【0031】本発明のMgO保護層を適用できるPDPの形式・方式は特に制限されず、MgO保護層を必要とするものであれば面放電型、対向型等のいずれにも適用することができる。また、PDPにおける誘電体・蛍光体の種類（材質）、形状等も特に制限なく、いずれのものにも適用することができる。

【0032】本発明のPDPは、上記MgO保護層の形成以外の点については公知のPDPの製造工程（フロント基板製造工程、リア基板製造工程、全体組立工程）に

【0033】

【発明の効果】本発明のMgO材料は、耐水和性が改善されており、大気中においてもきわめて安定していることから、MgO保護層の形成に非常に有利となり、PDPの生産性向上に大幅に寄与することができる。

【0034】また、本発明のMgO材料は、CaO等の不純物含有量が制御されていることから、紫外～可視域にわたり優れた透明性を発揮できる結果、PDPの蛍光体等の保護層として用いた場合には蛍光体の発光輝度を十分確保することができる。その結果として、従来のPDPに比して優れた性能をもつPDPを得ることが可能

となる。

【0035】このように、本発明によれば、耐水和性の改善により、PDPの生産性及び保護層の特性向上（ひいてはPDPの性能向上）を同時に達成することができ、PDPの工業的生産に大きく貢献することができる。

【0036】

【実施例】以下、実施例及び比較例を示し、本発明の特徴とするところをよりいっそう明確にする。

10 【0037】実施例1～10

表1に示す特性をもつようなMgO材料をそれぞれ製造した。

【0038】まず、MgO原料としてMgO純度98.3～99.9重量%の焼結MgO又はこれにCaO又はSiO<sub>2</sub>を添加したものをアーク炉中に充填した。この場合、いずれの実施例においても、通電中に少量ずつ原料を投入し、合計で4000kgをアーク炉に充填した。その後、炭素電極にてアーク電流を5時間通電し、溶融した。溶融後にMgOを約1週間かけて徐冷した。冷却して得られた塊状物において、炭素電極直下付近の溶融状態が良好でかつ結晶の発達した部分を選別し、これを電融MgOとして採取した。

【0039】採取した電融MgOは適正な粒度に破碎し、密度、平均結晶粒径及び遷移金属元素の含有量合計を測定した。その結果を表1に示す。なお、各物性は次のようにしてそれぞれ測定した。

【0040】（1）密度

電融MgOの密度はアルキメデス法により測定した。

【0041】（2）CaO及びSiO<sub>2</sub>の含有量

30 ICP（高周波誘導結合プラズマ）発光分光分析により測定した。

【0042】（3）平均結晶粒径

電融MgOを研磨した後、反射顕微鏡にて統計的な観察により測定した（すなわち、任意に選んだ100個の結晶をサンプリングし、それらの算術平均を平均結晶粒径とした）。

【0043】（4）遷移金属元素の含有量合計

40 遷移金属元素の含有量合計については、ICP発光分光分析法により各遷移金属元素の含有量を測定し、それらの合計を求めた。

【0044】

【表1】

7 実施例	MgO純度(重量%) 真空到達時間(min)	CaO (ppm)	SiO <sub>2</sub> (ppm)	CaO/SiO <sub>2</sub>	密度 (g/cm <sup>3</sup> )	平均結晶粒径 (μm)	8 遷移金属 元素(ppm)
1	99.8 10	720	530	1.36	3.53	1500	530
2	99.6 16	2910	740	3.93	3.54	1300	310
3	99.8 13	1720	660	2.61	3.53	1600	980
4	99.8 11	240	1160	0.21	3.53	800	470
5	99.7 11	140	1890	0.07	3.54	500	820
6	99.8 14	720	530	1.36	3.46	1500	560
7	99.9 11	190	60	3.17	3.49	6500	380
8	99.8 10	940	580	1.62	3.53	1500	320
9	99.8 11	700	500	1.40	3.53	540	700
10	99.95 7	140	50	2.80	3.55	-	220

【0045】なお、表1中、実施例10は単結晶MgO  
であるので、平均結晶粒径の表示はしない。

【0046】比較例1～20

実施例1と同様の方法により、表2に示す特性をもつMgO材料を製造し、実施例1と同様に密度、CaO及び\*20

\* SiO<sub>2</sub>の含有量、平均結晶粒径ならびに遷移金属元素  
の含有量合計を測定した。その結果を表2に示す。

【0047】

【表2】

比較例	MgO純度(重量%) 真空到達時間(min)	CaO (ppm)	SiO <sub>2</sub> (ppm)	CaO/SiO <sub>2</sub>	密度 (g/cm <sup>3</sup> )	平均結晶粒径 (μm)	遷移金属 元素(ppm)
1	99.8 45	690	160	4.31	3.53	1500	800
2	99.6 78	3200	340	9.41	3.54	1600	300
3	99.3 19	1300	2140	0.61	3.53	980	2350
4	99.8 48	1150	530	2.16	3.54	480	260
5	99.7 46	960	590	1.66	3.39	1200	1130
6	99.7 59	1230	500	2.46	3.31	1300	670
7	99.9 63	540	130	4.15	3.44	30	120
8	99.9 77	120	80	1.50	3.28	10	850
9	99.5 56	2200	1600	1.38	3.44	50	1130
10	99.95 29	290	40	7.25	3.55	-	70
11	99.9 23	660	270	2.44	3.41	1200	50
12	99.9 61	650	260	2.60	3.50	350	30
13	99.6 37	1100	2200	0.50	3.43	560	510
14	99.6 91	3100	800	3.88	3.47	850	40
15	99.9 78	710	160	4.73	3.51	1100	100
16	99.5 48	2200	1500	1.20	3.51	930	900
17	99.7 63	710	290	2.40	3.52	1100	1700
18	99.6 36	950	2400	0.40	3.47	730	510
19	99.5 65	3200	1280	2.50	3.46	1400	400
20	99.5 54	1200	640	1.88	3.47	470	2870

【0048】なお、表2中、比較例10は単結晶MgO  
であるので、平均結晶粒径の表示はしない。また、比較※50

※例7～9は微粉末原料を用いて焼成した焼結MgOであ

## 【0049】試験例1

実施例1～10及び比較例1～20で製造された各試料について、耐水和性試験を行った。

【0050】試験方法としては、まず各試料を粒径0.1～3mmの範囲に調整し、これを温度35℃・相対湿度80%の恒温・恒湿室に入れ、72時間放置後に取り出した。次いで、各試料を分子ターボポンプを有する真空チャンバーに入れ、真空度が $5 \times 10^{-6}$  Torr (目的真空度) になるまでの到達時間を測定した。この場

合、到達時間が短いものほど耐水和性に優れている。これらの結果(真空到達時間)を表1及び表2に示す。

【0051】以上の結果からも明らかなように、実施例のMgO材料は恒温・恒湿室中での水和が抑制ないしは防止されたため、真空チャンバー内で真空引きしたときの目的真空度に到達するまでに要する時間が比較例のものに比して非常に短くなっていることがわかる。これらのことから、本発明MgO材料がPDP用の保護層として非常に有用であることが明らかである。

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(54) MGO MATERIAL FOR PLASMA DISPLAY, ITS PRODUCTION AND PLASMA DISPLAY

(57)Abstract:

PROBLEM TO BE SOLVED: To produce an MgO material in which hydration is especially suppressed or prevented.

SOLUTION: This MgO material is the one used for a dielectric of a plasma display and/or a protective layer of a fluorescent substance and having (1)  $\geq 99.6$  wt.% MgO purity, (2)  $\leq 3,000$  ppm CaO content and  $\leq 2,000$  SiO<sub>2</sub> content in  $\leq 4$  weight ratio of the CaO/SiO<sub>2</sub>, (3)  $\geq 500$   $\mu$ m average crystal grain diameter and (4)  $\geq 3.45$  g/cm<sup>3</sup> density.

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CLAIMS

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[Claim(s)]

[Claim 1] the MgO ingredient used for the MgO protective layer of the dielectric of a plasma display, and/or a fluorescent substance -- it is -- 99.6 % of the weight or more of (1) MgO purity, (2) CaO content of 3000 ppm or less, and SiO<sub>2</sub> content of 2000 ppm or less -- \*\* \*\*\*\*\* -- and MgO ingredient for plasma displays characterized by being four or less CaO/SiO duplex quantitative ratio, 500 micrometers or more of diameters of (3) average crystal grain, and three or more (4) consistency 3.45 g/cm.

[Claim 2] The MgO ingredient for plasma displays according to claim 1 whose content sum total of Fe, Cr, V, Cu, nickel, Zr, and La is 1000 ppm or less as oxide.

[Claim 3] The manufacture approach characterized by electromelting the MgO raw material which is the manufacture approach of the MgO ingredient for plasma displays according to claim 1 or 2, and is 95 % of the weight or more of MgO purity.

[Claim 4] The plasma display with which the protective layer in either [ at least ] the dielectric of a plasma display or a fluorescent substance was formed considering the MgO ingredient for plasma displays according to claim 1 or 2 as a raw material.

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DETAILED DESCRIPTION

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[Detailed Description of the Invention]

[0001]

[Field of the Invention] This invention relates to the MgO ingredient for plasma displays used for the protective layer of the dielectric of a plasma display (the both sides of a monochrome plasma display and a color plasma display are included), and/or a fluorescent substance, its manufacture approach, and a plasma display.

[0002]

[Description of the Prior Art] In recent years, it excels in engine performance, such as brightness and luminous efficiency, and moreover, plasma displays (PDP) also with the enough display quality for a television display are marketed, and expectation is growing also in implementation-ization of long-awaited wall tapestry mold television.

[0003] PDP came [ on the Braun-tube type display (CRT) by the electron gun excitation currently used from the former, have left various problems to constraint of the Braun-tube volume and the display screen, brightness, etc., the display of the new method or structure which solves these problems was considered, and recent years came, and ] to attract attention at last.

[0004] Discharge (ultraviolet rays) excitation is carried out and the fluorescent substance which exists in each cel is made to emit light at PDP to the electron ray emitted from the electron gun carrying out excitation luminescence of the direct fluorescent substance in this conventional CRT.

[0005] PDP is divided roughly into counterelectrode structure (opposed type) and field discharge structure (field discharge mold) as the color display method. Counterelectrode structure applies a fluorescent substance near the electrode on one [ at least ] glass substrate, and excitation fluorescence is carried out by the UV irradiation by discharge. In this case, a fluorescent substance receives sputtering (ion bombardment) by impression of alternating voltage, and it is known that this will have big effect on the life of a fluorescent substance. Then, in order to protect a fluorescent substance from sputtering, the MgO protective layer is formed on the fluorescent substance as cathode material strong against this sputtering. On the other hand, with field discharge structure, the fluorescent substance which covered the MgO protective layer beforehand is applied on the rear-face glass substrate which does not receive an ion bombardment, and excitation luminescence is carried out by the UV irradiation accompanying discharge.

[0006] Thus, in PDP (especially AC mold), the MgO protective layer has played the very important role. However, in the actual condition, most researches on the property of the MgO ingredient (raw material) for forming this MgO protective layer are not made.

[0007]

[Problem(s) to be Solved by the Invention] Generally as the formation approach of the protective layer of PDP, electron beam vacuum deposition is adopted. MgO for which the electron beam was irradiated by the MgO raw material in the chamber maintained by the high vacuum condition and which volatilized is deposited on the dielectric beforehand formed on the glass substrate, or a fluorescent substance, and, specifically, a protective layer is formed by forming membranes.

[0008] However, from the conventional MgO raw material, there is a problem of being easy to hydrate the MgO (especially surface part) itself. That is, if the MgO raw material which the surface part hydrated is used as the raw material source of electron beam evaporation, long duration is taken to reach the object degree of vacuum, and it may have an adverse effect on productivity. On the other hand, although removing moisture from a MgO raw material beforehand is also considered, moisture is thoroughly unremovable, if it once hydrates,  $Mg(OH)_2$  is generated and it will not heat at the elevated temperature of 500 degrees C or more. For this reason, in the MgO raw material used especially for a PDP protective layer, a storage condition must be managed strictly and there is also constraint of a storage time.

[0009] On the other hand, a certain trouble may be brought to the quality of the MgO protective layer formed using such the raw material source itself.

[0010] Thus, in formation of a MgO protective layer, the hydration of MgO is the cause which causes lowering of the productivity of PDP, lifting of a manufacturing cost, or the debasement of PDP, and to cancel this is made into pressing need in manufacture of PDP.

[0011] Therefore, this invention is made in view of the actual condition of this conventional technique, and sets it as the main objects to offer the MgO ingredient by which hydration was controlled or prevented especially.

[0012]

[Means for Solving the Problem] As a result of repeating research wholeheartedly in view of the trouble of the conventional technique, this invention person finds out that the MgO ingredient which has a specific presentation and structure can attain the above-mentioned object, and came to complete this invention at last.

[0013] That is, this invention relates to the MgO ingredient for plasma displays, its following manufacture approach, and a following plasma display.

[0014] 1. the MgO ingredient used for the MgO protective layer of the dielectric of a plasma display, and/or a fluorescent substance -- it is -- 99.6 % of the weight or more of (1) MgO purity, (2) CaO content of 3000 ppm or less, and SiO<sub>2</sub> content of 2000 ppm or less -- \*\* \*\*\*\* -- and MgO ingredient for plasma displays characterized by being four or less CaO/SiO<sub>2</sub> duplex quantitative ratio, 500 micrometers or more of diameters of (3) average crystal grain, and three or more (4) consistency 3.45 g/cm.

[0015] 2. Manufacture approach characterized by electromelting MgO raw material which is manufacture approach of MgO ingredient for plasma displays publication, and is 95 % of the weight or more of MgO purity in the 1st term of the above.

[0016] 3. Plasma display with which protective layer in either [ at least ] dielectric of plasma display or fluorescent substance was formed in the 1st term of the above considering MgO ingredient for plasma displays of publication as raw material.

[0017] In addition, in this invention, "ppm" shows a weight rate (weight ppm).

[0018]

[Embodiment of the Invention] First, the MgO ingredient of this invention is a raw material (raw material source) used for the MgO protective layer of the dielectric of a plasma display, and/or a fluorescent substance.

[0019] What is necessary is just to usually make MgO purity of the MgO ingredient of this invention into 99.7 % of the weight or more preferably 99.6% of the weight or more. When MgO purity is less than 99.6 % of the weight, hydration-proof falls, or there is a possibility that the optical property of a protective coat may fall.

[0020] 3000 ppm or less (preferably 2000 ppm or less) and SiO<sub>2</sub> content of the CaO content in a MgO ingredient are usually 2000 ppm or less (preferably 1500 ppm or less). When a CaO content exceeds 3000 ppm, the hydration-proof of a MgO ingredient falls. When SiO<sub>2</sub> content exceeds 2000 ppm, when forming a PDP protective layer and Si<sup>4+</sup> ion permutes by Mg<sup>2+</sup> ion, a cation deficit is produced, the optical absorption edge of a MgO protective layer shifts to a long wavelength side (the MgO protective layer of a fluorescent substance absorbs the ultraviolet rays which are excitation light), and there is a possibility of reducing the luminescence brightness of a fluorescent substance.

[0021] Furthermore, in this invention, a CaO/SiO duplex quantitative ratio usually takes preferably or less for 3.5 four or less. When a CaO/SiO duplex quantitative ratio exceeds 4, the hydration-proof of a MgO ingredient falls.

[0022] Moreover, the diameter of average crystal grain in a MgO ingredient usually sets 500 micrometers or more to 1000 micrometers or more preferably. The consistency in a MgO ingredient is usually preferably made into three or more 3.48 g/cm three or more 3.45 g/cm. When the diameter of average crystal grain is less than 500 micrometers, and when consistencies are less than three 3.45 g/cm, a touch area with the moisture in atmospheric air becomes large as a whole, and hydration-proof may be reduced. The diameter of average crystal grain can control for example, SiO<sub>2</sub> content, or can adjust it suitably by adjustment (it is melting in the big large-sized electromelting furnace of heat capacity) of the cooling rate after electromelting etc. Moreover, the above-mentioned consistency can be suitably adjusted by sorting of extent which fuses a raw material, and a electromelting article etc.

[0023] In addition, especially the upper limit of the diameter of average crystal grain is not limited, therefore can also use a MgO single crystal. On the other hand, it is not restricted especially about the upper limit of a consistency, either, but can raise to theoretical density (3.58 g/cm<sup>3</sup>).

[0024] it is desirable that the content sum total of Fe, Cr, V, Cu, nickel, Zr, and La usually sets 1000 ppm or less to 700 ppm or less as an oxide especially with the MgO ingredient of this invention as O<sub>3</sub>, CrO<sub>2</sub>, V<sub>2</sub>O<sub>5</sub>, and CuO, NiO, ZrO<sub>2</sub> and La<sub>2</sub>O<sub>3</sub> namely, Fe<sub>2</sub>. By controlling the content sum total of these elements (henceforth a "transition-metals element") in the above-mentioned range, the transparency by which the MgO protective layer especially formed using this MgO ingredient was stabilized over ultraviolet - a visible range can be discovered more effectively. In order to control the total amount of a transition-metals element in the above-mentioned range, it can carry out by choosing for example, a MgO raw material (presentation), or performing deironation treatment at the time of grinding of a MgO raw material etc. Deironation treatment can be carried out by processing by acid cleaning and the magnetic separation machine etc.

[0025] Especially although the manufacture approach of this invention MgO ingredient is not limited especially as long as the thing of above-mentioned within the limits is obtained fundamentally, it is desirable to manufacture by electromelting the MgO raw material which is 95 % of the weight or more of MgO purity. According to this electromelting method, compared with a sintering process, it is advantageous at the point that it is easy to control within the limits of the above-mentioned predetermined diameter of average crystal grain, and a consistency, and the MgO raw material of about 95 % of the weight of MgO purity can moreover be used.

[0026] As for especially the MgO purity of a MgO raw material, it is usually desirable that it is 98 % of the weight or more 95% of the weight or more. By this invention approach which adopts the electromelting method, since an impurity can evaporate or only Electromelting MgO can be substantially sorted out comparatively easily from an impurity moving and being unevenly distributed in the periphery of Electromelting MgO with electromelting (melting), it becomes possible to obtain MgO with purity higher than a raw material MgO. And by repeating a series of processes of electromelting and sorting for the electromelting MgO sorted out further, high grade-ization can be attained more and such a mode is also included by the manufacture approach of this invention. Therefore, when using what has MgO purity comparatively low as a raw material MgO, it is desirable to repeat and carry out the above-mentioned process until it becomes predetermined MgO purity and a predetermined CaO content, SiO<sub>2</sub> content, and a CaO/SiO duplex quantitative ratio.

[0027] In the manufacture approach of this invention, a CaO content, SiO<sub>2</sub> content, and a CaO/SiO duplex quantitative ratio are controllable by repeating for example, the above-mentioned process, and also it is controllable by adding CaO and SiO<sub>2</sub> grade positively. These [ CaO ] and SiO<sub>2</sub> grade can use a commercial item as it is.

[0028] The operating condition of the electromelting in the manufacture approach of this invention, electromelting equipment, etc. can adopt conditions, equipment, etc. which are adopted by the well-known electromelting approach as it is. For example, what is necessary is to cool, after filling up an arc furnace with a predetermined MgO raw material, fusing by energizing an arc current in a carbon

electrode and completing melting (annealing), to sort out Electromelting MgO and just to take out from the obtained massive object.

[0029] A MgO protective layer [ in / in PDP of this invention / either / at least / the dielectric of a plasma display or a fluorescent substance ] is formed considering the MgO ingredient for this invention plasma displays as a raw material (raw material source). After the formation approach of a MgO protective layer can apply well-known thin film coating technology as it is, for example, forms a dielectric or a fluorescent substance layer, it can produce a MgO thin film using approaches, such as electron beam vacuum deposition.

[0030] What is necessary is just to set up MgO protection layer thickness suitably according to the application of the format and method of PDP, and the final products (a dielectric, fluorescent substance, etc.) for formation of a protective layer etc.

[0031] Especially the format and method of PDP which can apply the MgO protective layer of this invention are not restricted, but if it needs a MgO protective layer, it is applicable to both a field discharge mold, an opposed type, etc. Moreover, the class (construction material) of the dielectric and fluorescent substance in PDP, especially a configuration, etc. do not have a limit, and can be applied to anything.

[0032] What is necessary is just to manufacture PDP of this invention according to the well-known production process of PDP about points other than formation of the above-mentioned MgO protective layer (like a front substrate production process, a rear substrate production process, and a whole erector).

[0033]

[Effect of the Invention] Since hydration-proof is improved and the MgO ingredient of this invention is extremely stable in atmospheric air, it becomes very advantageous to formation of a MgO protective layer, and can be substantially contributed to the productivity drive of PDP.

[0034] Moreover, since impurity contents, such as CaO, are controlled, as a result of being able to demonstrate the transparency which was excellent over ultraviolet - a visible range, the MgO ingredient of this invention can secure the luminescence brightness of a fluorescent substance enough, when it uses as protective layers, such as a fluorescent substance of PDP. It becomes possible to obtain PDP with the engine performance which was excellent as the result as compared with the conventional PDP.

[0035] Thus, according to this invention, by improvement of hydration-proof, the productivity of PDP and the improvement in a property of a protective layer (as a result, improvement in the engine performance of PDP) can be attained simultaneously, and it can contribute to industrial production of PDP greatly.

[0036]

[Example] Hereafter, an example and the example of a comparison are shown and the place by which it is characterized [ of this invention ] is clarified further.

[0037] A MgO ingredient which has the property shown in one to example 10 table 1 was manufactured, respectively.

[0038] First, it was filled up with what added CaO or SiO<sub>2</sub> as a MgO raw material to the sintering MgO of 98.3 - 99.9 % of the weight of MgO purity, or this into the arc furnace. In this case, also in which example, the small quantity [ every ] raw material was thrown in during energization, and the arc furnace was filled up with 4000kg in total. Then, the arc current was energized for 5 hours and fused in the carbon electrode. It cooled slowly after melting, having applied it for about one week. The massive object cooled and obtained set, the melting condition near directly under [ carbon-electrode ] was good, and sorted out the part from which the crystal developed, and this was extracted as electromelting MgO.

[0039] The extracted electromelting MgO was crushed to a proper grain size, and measured the content sum total of a consistency, the diameter of average crystal grain, and a transition-metals element. The result is shown in a table 1. In addition, each physical properties were measured as follows, respectively.

[0040] (1) the consistency of the consistency electromelting MgO -- Archimedes -- it measured by law.

[0041] (2) It measured by the content ICP (RF inductively coupled plasma) emission spectral analysis of

CaO and SiO<sub>2</sub>.

[0042] (3) After grinding the diameter electromelting MgO of average crystal grain, it measured by statistical observation with the reflecting microscope (that is, 100 crystals chosen as arbitration were sampled and those arithmetic means were made into the diameter of average crystal grain).

[0043] (4) About the content sum total of the content sum total transition-metals element of a transition-metals element, the content of each transition-metals element was measured by ICP emission spectrochemical analysis, and those sum totals were searched for.

[0044]

[A table 1]

実施例	MgO純度(重量%)	CaO	SiO <sub>2</sub>	CaO/SiO <sub>2</sub>	密度	平均結晶粒径	遷移金属
	真空製造時間(min)	(ppm)	(ppm)		(g/cm <sup>3</sup> )	(μm)	元素(ppm)
1	99.8 10	720	530	1.36	3.53	1500	530
2	99.8 16	2910	740	3.93	3.64	1300	810
3	99.6 13	1720	660	2.61	3.53	1600	980
4	99.8 11	240	1160	0.21	3.53	800	470
5	99.7 11	140	1890	0.07	3.54	500	820
6	99.8 14	720	530	1.36	3.46	1500	560
7	99.9 11	190	80	3.17	3.49	8500	980
8	99.8 10	940	580	1.62	3.53	1500	320
9	99.8 11	700	500	1.40	3.53	540	700
10	99.95 7	140	50	2.80	3.55	—	220

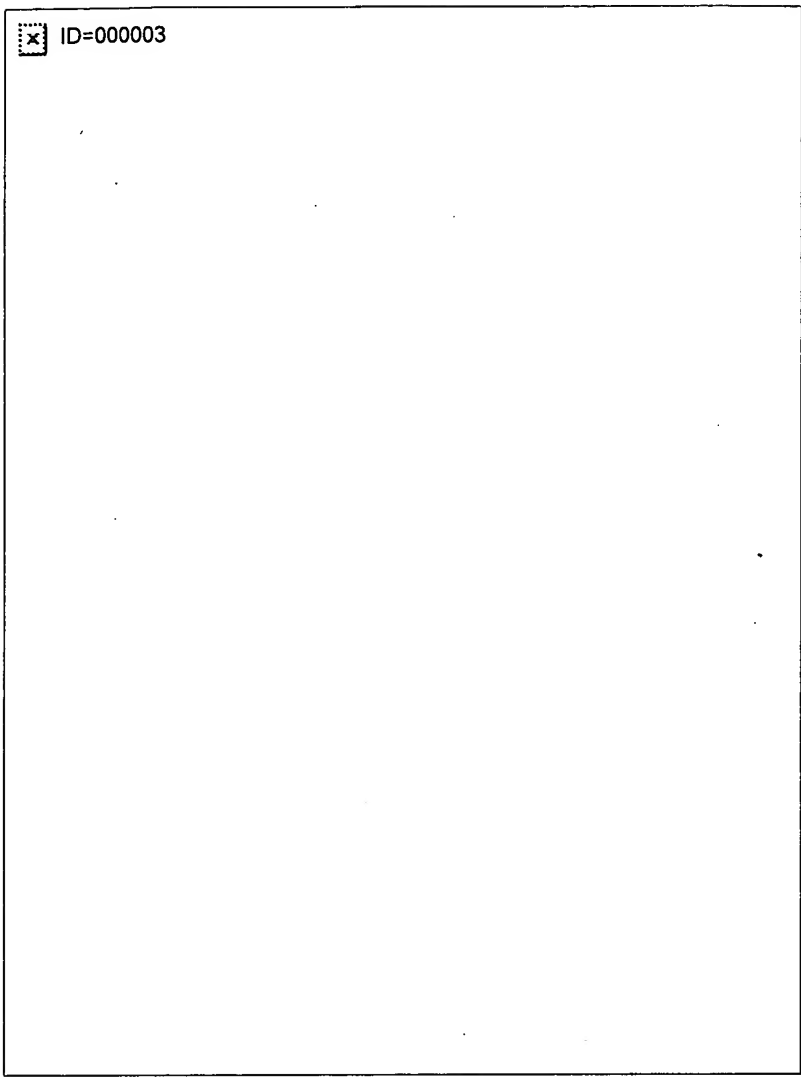
[0045] In addition, among a table 1, since an example 10 is a single crystal MgO, the diameter of average crystal grain is not indicated.

[0046] By the same approach as one to example of comparison 20 example 1, the MgO ingredient with the property shown in a table 2 was manufactured, and the content sum total of a consistency, the content of CaO and SiO<sub>2</sub>, the diameter of average crystal grain, and a transition-metals element was measured like the example 1. The result is shown in a table 2.

[0047]

[A table 2]



ID=000003

[0048] In addition, among a table 2, since the example 10 of a comparison is a single crystal MgO, the diameter of average crystal grain is not indicated. Moreover, the examples 7-9 of a comparison are the sintering MgO calcinated using the impalpable powder raw material.

[0049] The waterproof sum sex test was performed about each sample manufactured in example of trial 1 examples 1-10, and the examples 1-20 of a comparison.

[0050] as a test method -- first -- each sample -- the range of 0.1-3mm particle size -- adjusting -- this -- the constant temperature of the temperature of 35 degrees C, and 80% of relative humidity -- it put into - constant humidity room, and took out after 72-hour neglect. Subsequently, each sample was paid to the vacuum chamber which has a molecule turbine pump, and time of concentration until a degree of vacuum is set to  $5 \times 10^{-6}$  Torr (object degree of vacuum) was measured. In this case, the thing which has short time of concentration is excellent in hydration-proof. These results (vacuum time of concentration) are shown in a table 1 and a table 2.

[0051] clear also from the above result -- as -- the MgO ingredient of an example -- constant temperature -- it turns out that it can fold and the time amount taken to reach the object degree of vacuum when [ at which vacuum suction was carried out within the vacuum chamber ] the hydration in the inside of - constant humidity room is controlled or prevented is very short as compared with the thing of the example of a comparison. It is clear from these things that this invention MgO ingredient is dramatically useful as a protective layer for PDP.

[Translation done.]